Barnes.

A Study Of The Heat Treatment Of Steel For Shafting.

A STUDY OF THE HEAT TREATMENT OF STEEL FOR SHAFTING

BY

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THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Otis Avery Barnes

ENTITLED Heat Treatment of Steel for Shafting

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Bachelor of Science

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The author wishes to take this opportunity to express his appreciation and thanks to his friend Dr.D.F.McFarland for the encouragement and many suggestions so willingly and courteously extended.

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Heat Treatment of Steel for Shafting

Introduction. The heat treatment of metals for industrial purposes is a subject which, until the last fifteen years, has not received from scientists the attention which its importance deserves. Within this limit of time, investigators have proven conclusively the advantages of scientifically heat-treated metals, over those which were untreated and those treated by rule of thumb methods.

It is for this reason that the present problem concerning the correct heat treatment of steel was undertaken. The heat treatment of steel for shafting was chosen, because of the fact that the Engineers' Society of Western Pennsylvania considered this of sufficient importance, to offer it as the annual competitive problem to seniors doing research work in Metallurgical Engineering. The statement of their problem is as follows:

"Determine by chemical, physical or microscopical, or a combination of any or all three, the heat treatment which will place steel, similar to the sample submitted, in the best condition for use as shafting or for similar purposes. Material is of the grade ordinarily furnished for heavy duty machinery shafting or steam railroad axles. This material is subjected to vibratory stresses and shocks, and the experiment should therefore indicate as fully as possible under what conditions the material is best able to resist them. Complete details of

experiments, including photomicrographs, should be submitted, and detailed reasons for the conclusions which have been drawn.

"Reference to any literature on the subject of failure of material from fatigue, also a discussion of the theory sometimes advanced that this is due to "Cold crystallization", should be given."

It is to be hoped that the results of such a competitive investigation will be of some value, not only to those doing the work, but also to those engaged in the manufacture and manipulation of steel for shafting.

Standard Specifications. As a first step in this investigation, it is proper to inquire what are the existing standards for steel which is to be used for making shafting. Standard specifications for such material have been drawn up by the American Society for Testing Materials, and are published in the 1915 Yearbook of that Society (pp. 115 - 120). The following extracts are from these specifications.

Table I

The steel shall conform to the following chemical composition:

Carbon	0.35 to 0.60%
Manganese	0.40 to 0.70%
Phosphorus	not over 0.05%
Sulphur	not over 0.05%

Table II

The steel shall possess the following minimum tensile strength:

Tensile strength, lb. per sq. in.	85,000
Elastic limit, lb. per sq. in.	50,000
Elongation in 2 in., not under	20.5%
Reduction of area, not under	39%

The elastic limit shall be determined by means of an extensometer.

Tension test specimens shall be of the standard 2 in. by 1/2 in. form and dimensions.

Plan of work. The scheme of work planned was first to analyze the sample furnished, and then obtain material of approximately the same composition sufficient for the tests which are to be used. Duplicate analyses were to be run in each case, after which cooling curves were to be taken and the critical points of the steel determined. Knowing these critical points, the standard test pieces which had been turned out were to be given the desired heat treatments, and the tensile tests then applied. Cross sections were to be prepared of all specimens and their microscopical structure studied, after which the hardness of each was to be determined by the Brinell method.

Experimental Part

Preliminary Investigation. Before beginning the study of the steel sample submitted for the competition, a series of

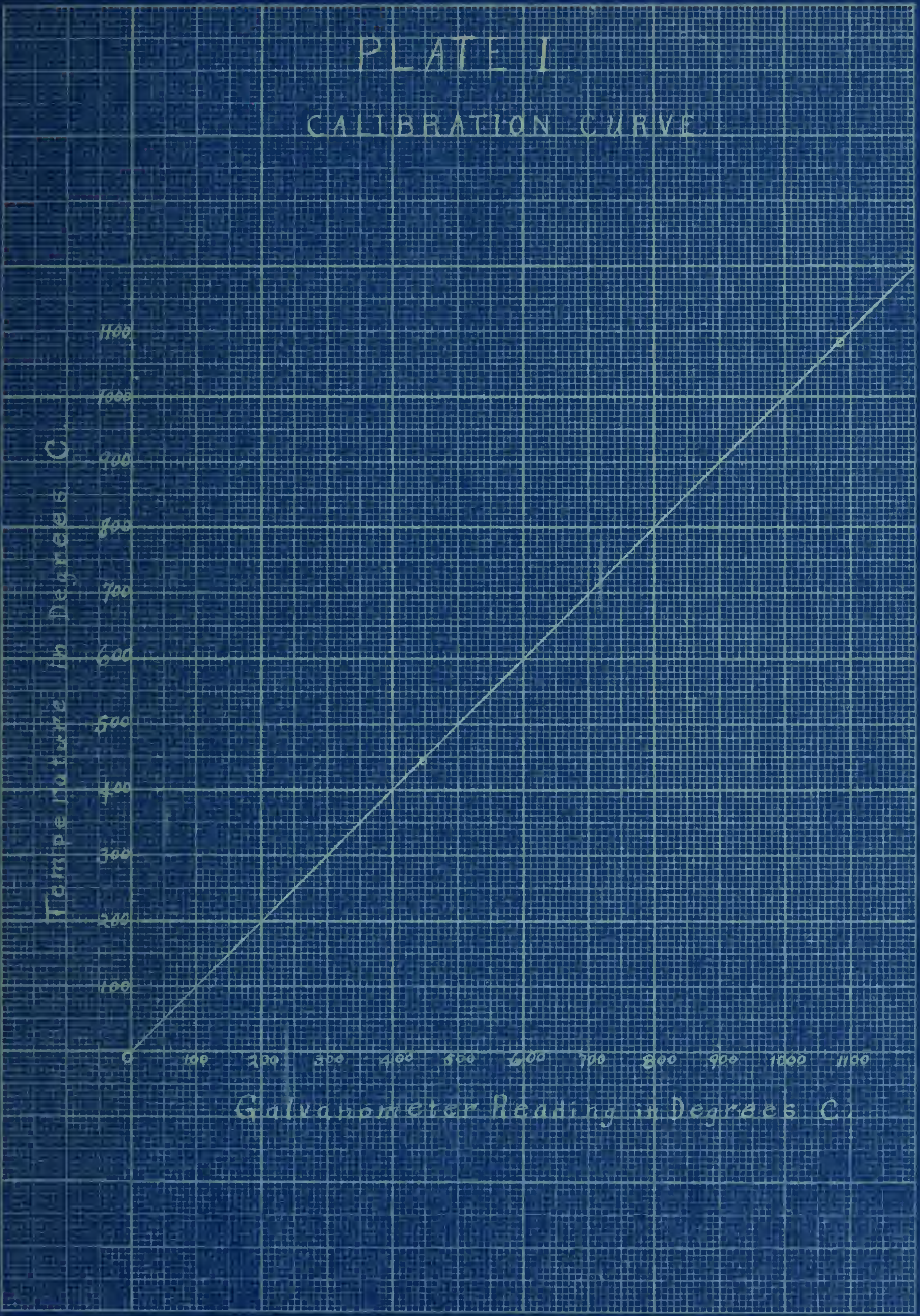
preliminary experiments was made with steel of known composition, in order to determine the best methods of working and the relative efficiency of the apparatus used. For this purpose, steel of known carbon composition was obtained from the Theoretical and Applied Mechanics Laboratory of the University of Illinois and given a thermal study.

Calibration of Pyrometer. A LeChatelier thermo-electric pyrometer was used with a Pt, Pt-Rh couple. This pyrometer was calibrated in the usual manner, by determining the freezing point of pure copper under reducing conditions and the boiling point of sulphur. The cold junction was maintained at 0° C thruout the experiment. Using these two points, a curve was constructed, Plate I, with readings of the pyrometer as abscissae and actual temperatures as ordinates. By means of this curve, it was possible to determine quickly the actual temperature from a reading on the millivoltmeter.

Cooling Curves. A cooling curve was now taken of the steel under preliminary investigation. The hot junction was inserted into a small hole drilled into the end of the steel, the steel heated up to about 1000° C in a platinum resistance furnace, the furnace closed tightly, the current shut off, and the heated steel allowed to cool as slowly as possible. Readings of the galvanometer were taken every half minute. These readings were then plotted as ordinates directly against time as abscissae. The value of such a curve is, that it indicates the temperature at which each change of state takes place, and the

PLATE I

CALIBRATION CURVE



range over which each development of heat continues.

Preparation of Microscopical Sections. Specimens of the auxiliary steel were now annealed by heating to just above the first critical point Ar_1, and allowed to cool as slowly as possible. Thin sections of about 1/4 in. thickness were cut off by means of a power hack saw. The surfaces were then prepared for polishing by rubbing on emery paper. In this case French emery paper (Hubert - - - 1, 0, 00, 000, and 0000) was used. Much time was saved by use of a machine, by means of which the specimens were held on the emery paper attached to rapidly rotating discs. In changing from one grade of paper to the next finer, the sections were held so that the new scratches were formed perpendicular to the old. The final polishing was done by holding the sections on a rotating disc covered with cloth, and wet with an aqueous suspension of rouge.

In order to render the structure clearly visible etching was necessary. This was accomplished by use of picric acid solution. A 1 % alcohclic solution was employed. About thirty seconds etching was found sufficient to develop the structure, after which the surface was washed with alcohol and dried by air without a cloth. This latter precaution prevented the tarnishing of the specimen by picric acid.

Microscopic Examination. For examination of the internal structure of the prepared specimen, a Leitz Micro-metallagraph was used with Huyghenian eyepieces and Achromatic objectives. At first a small electric arc was used for illumina-

tion, but on account of sputtering and variation a 250 watt Mazda nitrogen filled lamp was introduced. It gave satisfactory results but less illumination.

Furnace. Considerable doubt was first expressed as to whether it would be possible to obtain a furnace, which would give a distribution of temperature in the interior sufficiently uniform to insure that all parts of the specimen under examination would receive the same thermal treatment. After considerable discussion it was finally decided to run a preliminary investigation in the platinum resistance furnace.

A specimen of steel of nearly the length of the furnace was used. First, a section was taken from each end of the steel and suitably labeled. Then the steel was annealed by heating up to just above its first critical point Ar1, and allowed to cool slowly. This latter treatment insured a uniform internal structure thruout. Sections were again taken from each end and labeled. The steel was now heated up to just above its recalescent point, and then suddenly cooled by quenching in ice water. Sections were taken from each end as before, and the six specimens after polishing and etching were examined under the microscope. Each pair showed exactly the same internal structure, showing that the platinum resistance furnace would give satisfactory conditions for the thermal study of small masses of metal.

Research

Analysis of Steel Furnished. (Steel A). Analyses were made to determine the composition of the steel sample furnished by the Engineers' Society of Western Pennsylvania. This steel will hereafter be referred to as steel A.

Carbon. The determination of carbon was made by direct combustion using a silica tube contained in an electric furnace. The carbon dioxide was dissolved in potassium hydroxide 50% solution. A counter weight was used and care was taken to always weigh the absorbing bulb under the same conditions. An alundum boat was used for holding the borings.

Phosphorus. Phosphorus was determined by first dissolving in nitric acid, and subsequent oxidation of carbonaceous matter by saturated potassium permanganate solution. Any precipitated manganese dioxide was redissolved, and the phosphorus precipitated in dilute nitric acid solution as ammonium phosphomolybdate. This yellow precipitate was dissolved in standard alkali ($N a O H$) and the excess determined by standard acid ($H N O_3$). Phenolphtalein was used as an indicator. The solutions were standardized against a standard iron sample, sent out by the Bureau of Standards.

Sulphur. Sulphur was determined by first removing the silica by evaporation to dryness, baking, and subsequent digestion in hot dilute $H Cl$. The silica was filtered off and sulphur precipitated by $Ba Cl_2$ and weighed as $Ba S O_4$.

Manganese. Manganese was determined volumetrically

by oxidation in nitric acid solution of the manganous salts to permanganic acid by sodium bismuthate. The permanganic acid after filtration was titrated against standard sodium arsenite solution.

<u>Silicon</u>. Silicon was determined by dissolving in nitro-sulphuric acid with evaporation to white fumes to destroy all carbonaceous matter. On dilution and addition of H Cl the silicic acid was precipitated.

Samples of 0.4 carbon steel were now obtained from the Theoretical and Applied Mechanics Laboratory and analyzed in order to determine whether they were of the same composition as the sample furnished by the Engineers' Society of Western Pennsylvania. This steel will hereafter be referred to as Steel B. Analyses similar to the preceding showed this new material to have approximately the desired composition.

Table III

	Steel A	Steel B
	Steel from Engineers' Society	Steel from T. & A. M. Lab.
Carbon	0.40%	0.45%
Manganese	0.30%	0.64%
Phosphorus	0.01%	0.01%
Sulphur	0.04%	0.04%
Silicon	Traces	Traces

For the purposes of this investigation these analyses were considered sufficiently similar to permit of the use of this new steel for heat treatment. Sufficient material for the tests of this investigation was then obtained from the Theoretical and Applied Mechanics Laboratory. Both steels give values which fulfill the Standard Specifications for Chemical Composition given in Table I, page 2.

Cooling Curves. The cooling curves of the two steels were taken, one being on the original sample, and a second of the steel from the Theoretical and Applied Mechanics Laboratory.

The original sample gave an arrest in the cooling curve, Plate 2, at 693° (Ar_1) and at 729° (Ar_2). The specimen from the mechanical laboratory gave only one point, Plate 3, at 719° (Ar_1). It seems probable therefore that a lowering of the manganese content, causes a raising of the recalescent point, since the manganese per cent, Table 3, was the only analysis which showed any considerable variation in the two steels.

Heat Treatment. Standard two inch test specimens were now turned out according to specifications, Plate 7, and threaded on each end.

Table IV

These were then treated as follows:

Specimen No.	Treatment
1	Steel A as received
2	Steel A annealed from 900°

Table IV (cont.)

Specimen No.	Treatment
Steel B from Theoretical and Applied Mechanics Laboratory	
3	Annealed from 900°
4	Annealed from 900°; Quenched in ice water 750°; Tempered 670°.
5	Quenched in ice water from 750°
6	Quenched in ice water from 800°
7	Quenched in ice water from 900°
8	Quenched in ice water from 1000°

In carrying out the heatings the temperatures were controlled by the above described LeChatelier pyrometer.

The reasons for choosing these temperatures can best be explained by reference to the Roberts-Austin Roozeboom diagram shown below.

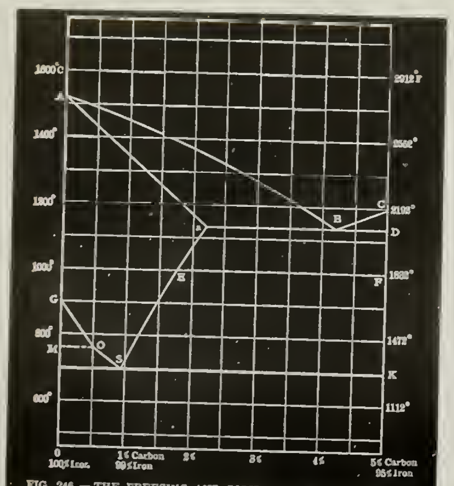


FIG. 246.—THE FREEZING AND SOLID DECOMPOSITION CURVES OF THE IRON-CARBON ALLOYS.

PLATE II.

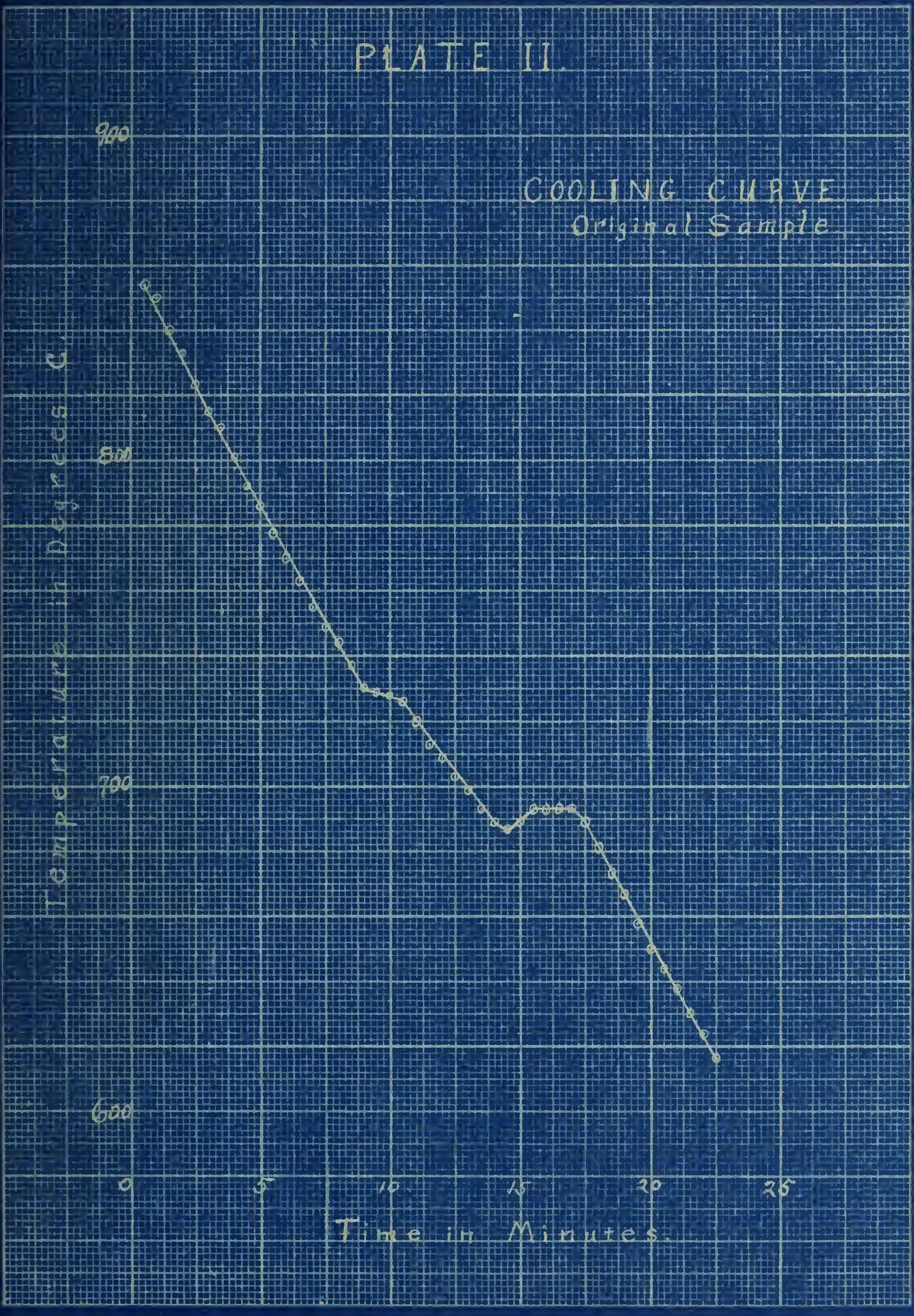
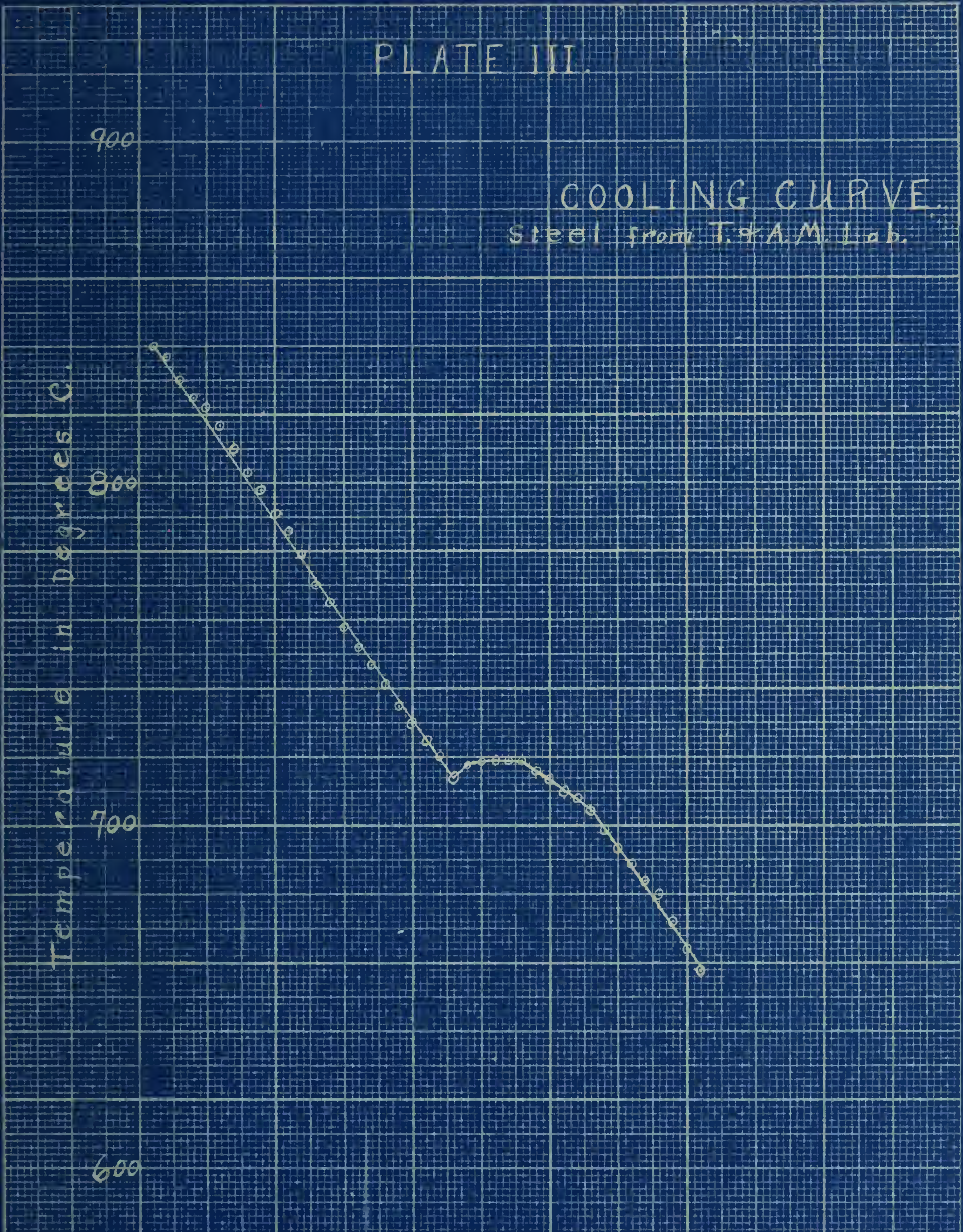

COOLING CURVE
Original Sample
Temperature in Degrees C.
900
800
700
600
0
5
10
15
20
25
Time in Minutes.

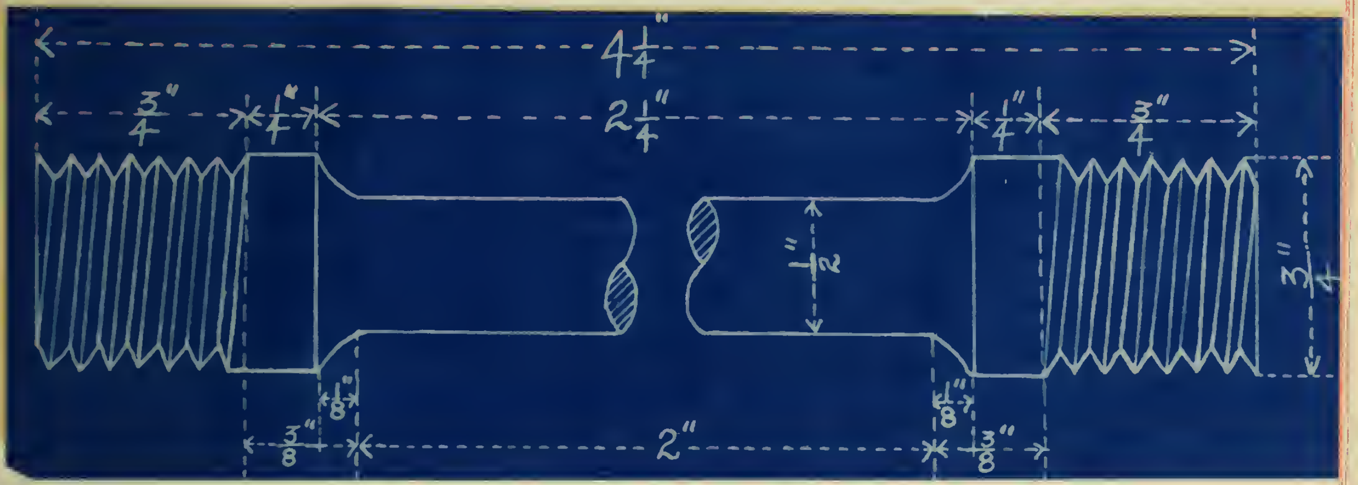
PLATE III.



COOLING CURVE.
Steel from T.&A.M. Lab.

PLATE VII.

Specification for Standard Two Inch Test Specimen.



With 0.5% carbon steel we should have two critical points; first at Ar_1 or about 690° where complete precipitation of pearlite occurs; second at Ar_2 or about 780° where ferrite begins to precipitate out.

The steels were annealed from 900° in order to allow uniform crystallization to take place. This also gave the crystals sufficient time to assume their natural size and shape. It destroyed any former crystallization due to any external treatment while passing through the Ar_1 or Ar_2 development.

The steels were quenched at 750° or just above Ar_1 and below Ar_2. This allowed us to study the internal structure and physical strength between the two points Ar_1 and Ar_2. Steel 4 was tempered from 670° in order to remove any internal stresses or strains caused by the sudden cooling. The other points were arbitrarily chosen without reference to the diagram. Higher temperatures were used since the author wished to study the effect of such temperatures on the physical and microscopical properties of the steel.

Tension Tests. These test pieces were now taken over to the Theoretical and Applied Mechanics Laboratory, and placed in the Riehle 100,000 lb. testing machine, the speed of head up to yield point being 0.1 inch per minute.

Extensometer tests were run on Specimens Nos, 1, 4, and 6. The clamps slipped on Nos. 2, 3, and 5 ruining the extensometer readings. In Nos. 7 and 8 it was thought best to dispense with the extensometer altogether, since the sudden

breaking of the metal endangered the instrument.

Table V

The following data was obtained:

Specimen No.	Load at Y.P. lb. per sq. in.	Load at Ult. lb. per sq. in.	Elongation %	Reduction Area %
1	51690	87700	25.0	36.0
2	44750	81400	18.7	35.4
3	47500	73300	16.9	32.0
4	59000	112200	37.5	53.6
5	#	146000	.00	4.5
6	#	149000	000	2.1
7	#	159000	.00	0.9
8	#	179000	000	5.0

That point in the stress diagram, where the unit stress ceases to be proportional to the unit elongation gives us the elastic limit. From the extensometer readings, curves were plotted, and we find that the sample as received (Plate 4) has an elastic limit of 42500 lb. per sq. in. The specimen which was annealed, quenched, and tempered has an elastic limit, (Plate 5) of 52,000 lb. Number 6 or the one quenched at 800° has an elastic limit (Plate 6) of 101,000 lbs.

By comparison with Table 2, page 3, we find that test piece No. 4 is the only one which fulfills all the physical tests for shafting given in the Standard Requirements.

Not determined for the above mentioned reasons.

PLATE IV.

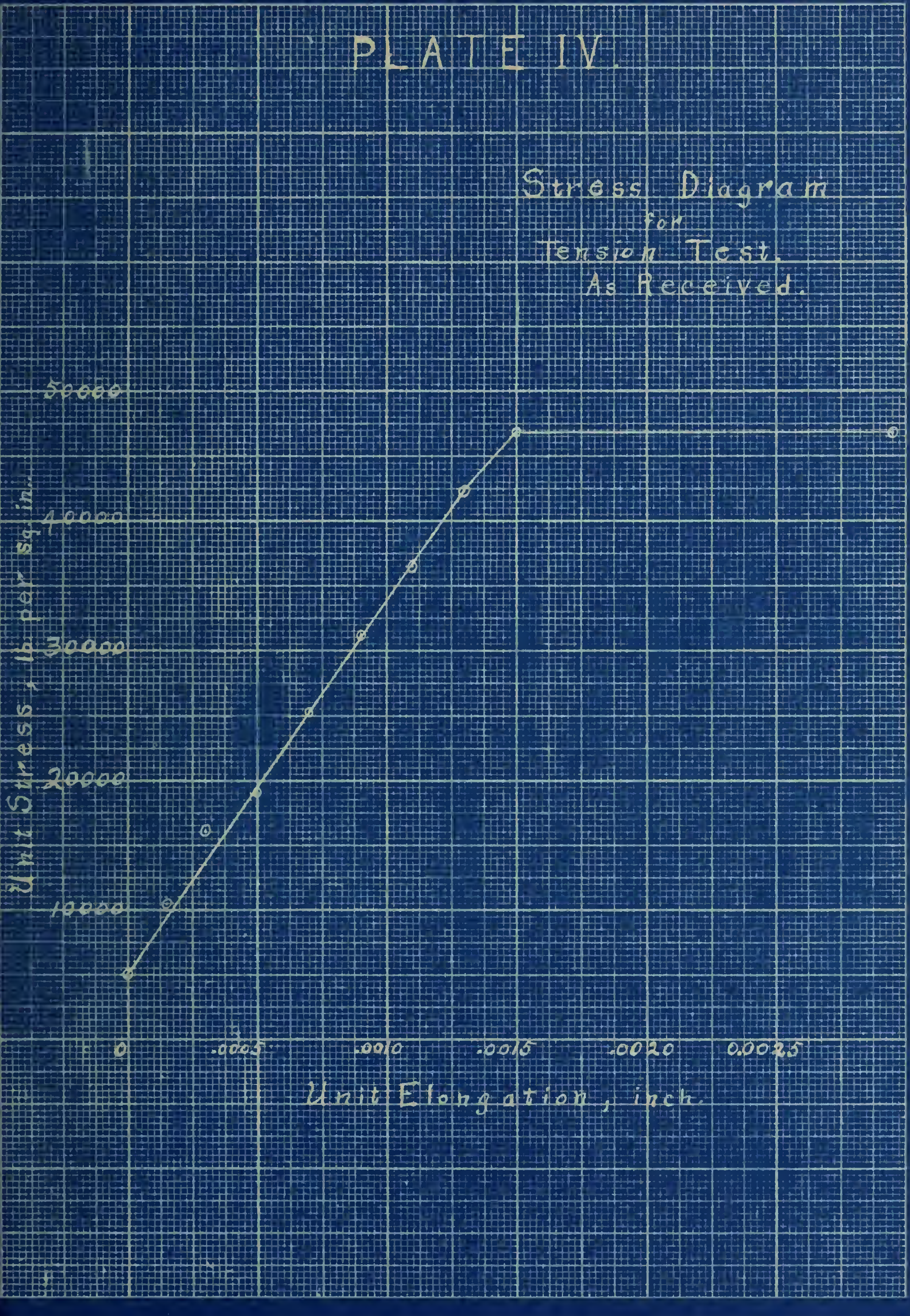

Stress Diagram
for
Tension Test.
As Received.
50000
40000
30000
20000
10000
0
.0005
.0010
.0015
.0020
0.0025
Unit Stress, lb. per sq. in.
Unit Elongation, inch.

PLATE V

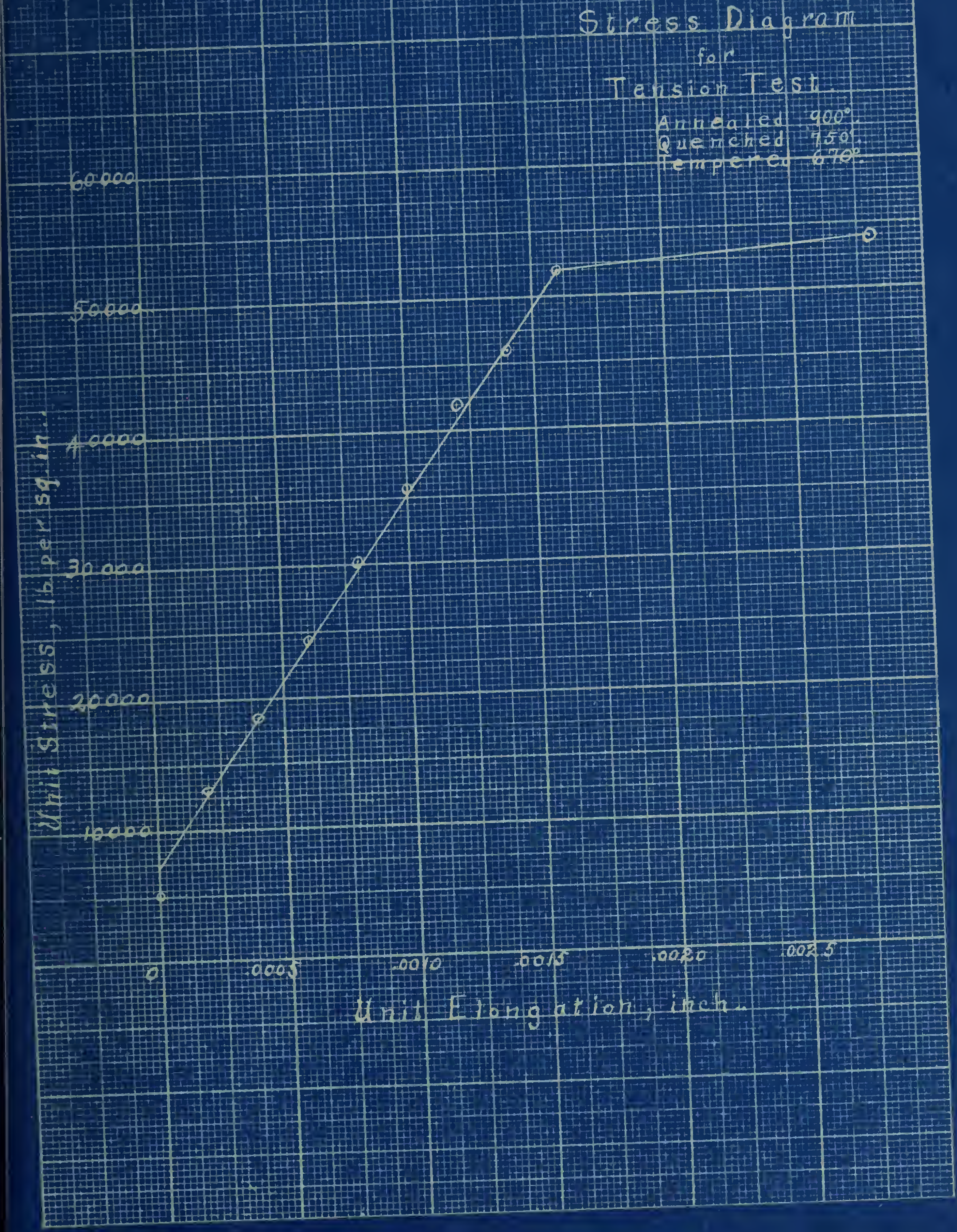
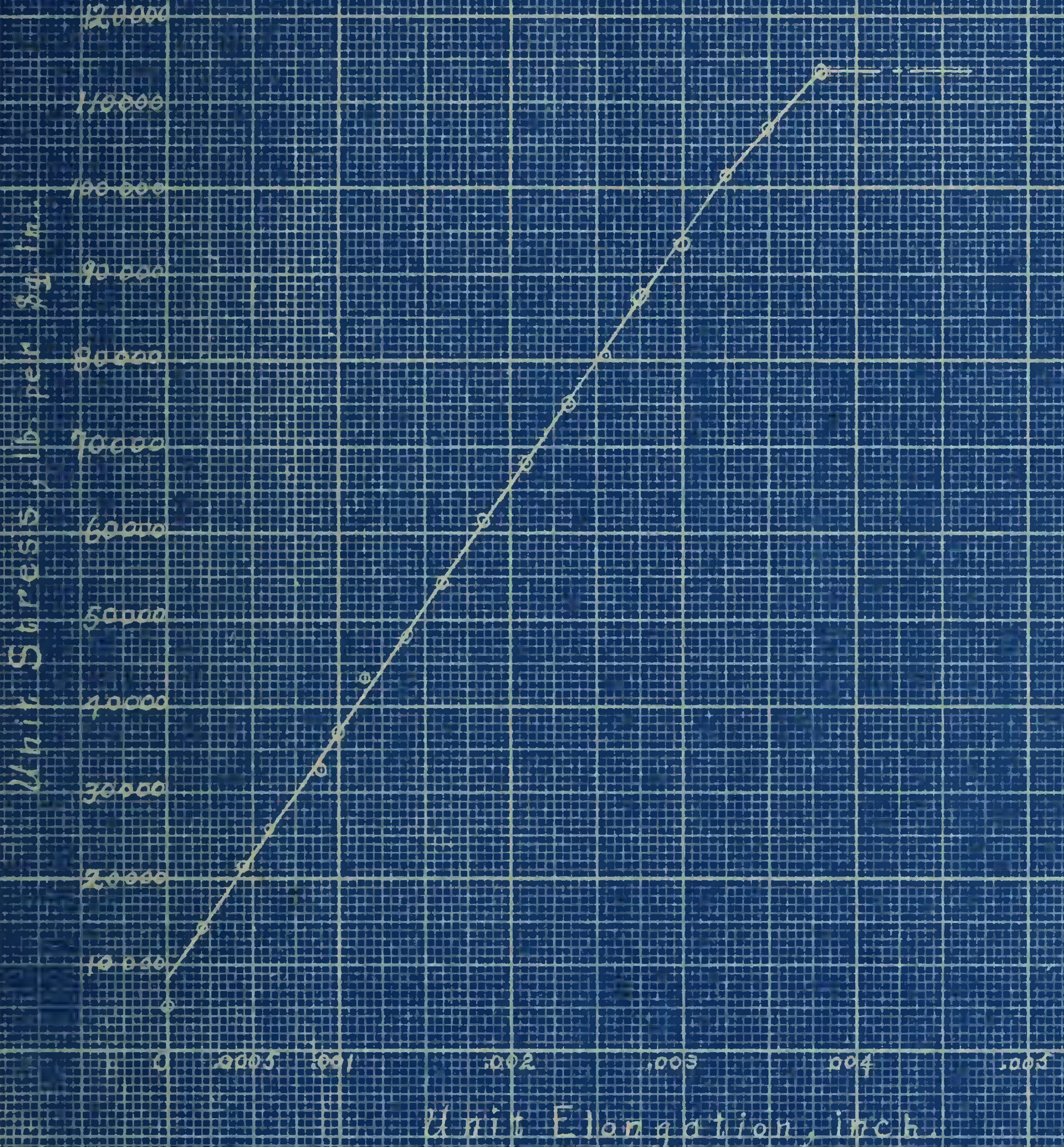

Stress Diagram
for
Tension Test
Annealed 900°.
Quenched 750°.
Tempered 670°.
60000
50000
40000
30000
20000
10000
Unit Stress, lb. per sq. in.
0
.0005
.0010
.0015
.0020
.0025
Unit Elongation, inch.

PLATE VI.

Stress Diagram
for
Tension Test.
Quenched 800°



Microscopical Examination. The results obtained in the tensile tests were supplemented by hardness measurements. First cross sections of the different specimens were prepared for microscopic study, in order to determine the internal structure, physical arrangement of the crystals, inherent defects if any, distribution of the various metalloids, relative proportion of constituents present, grain size, and the influence of the various heat treatments.

See Plate 8.

Fig. 1, Steel A. Sample as received. Shows a white network with occasional white patches of ferrite, and a much larger proportion of pearlite.

Fig. 2, Steel A. Sample annealed at 900° C. Shows complete recrystallization. Structure has more uniform sized crystals than in Fig. 1. Has light areas of ferrite and dark areas of pearlite. Shows considerable diffusion.

Figs. 3A and 3B, Steel B. T. & A. M. steel annealed at 900°. Shows pearlite structure, but very fine grained; probably sorbite.

Fig. 4, Steel B. Annealed from 900°, quenched at 750° in ice water, tempered from 670°. Has white areas of ferrite with some darker sorbitic structure, and dark patches of pearlite.

See Plate 9.

Fig. 5, Steel B. Quenched at 750° C in ice water. Shows polygonal formation of ferrite crystals filled in with

PLATE VIII.

Steel A--Sample from Eng.Soc.. Steel B--From T.& A.M.Lab..

Fig.1.
Steel A as received.
Etched 80 Seconds.
Exposed 90 Seconds.
X 250.

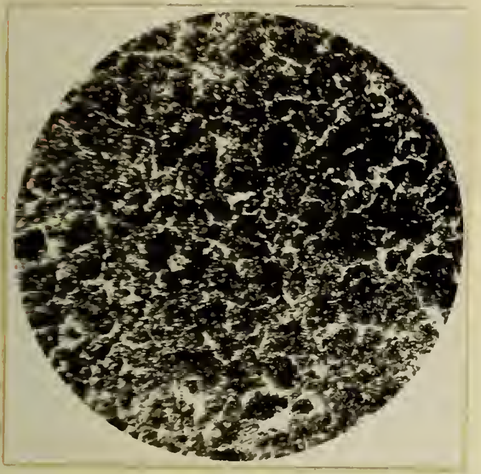


Fig.2.
Steel A annealed.
Etched 50 Seconds.
Exposed 120 Seconds.
X 250.

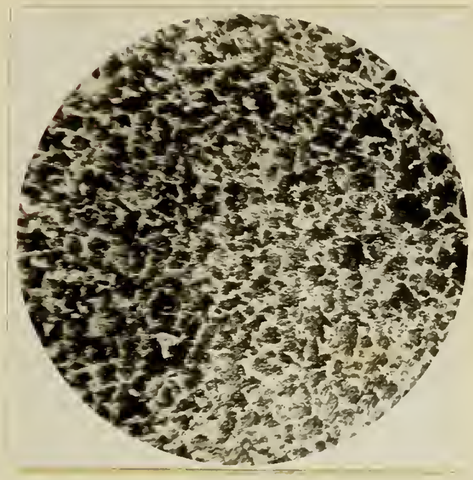


Fig.4.
Steel B;annealed 900°;quenched 750°;tempered 670°.
Etched 50 Seconds.
Exposed 90 Seconds.
X 250.

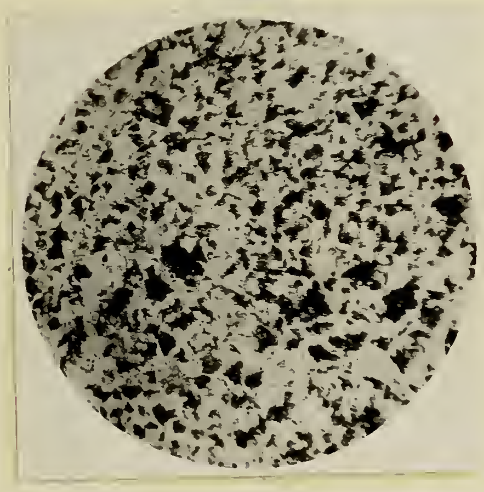


Fig.3a.
Steel B annealed.
Etched 50 Seconds.
Exposed 130 Seconds.
X 250.

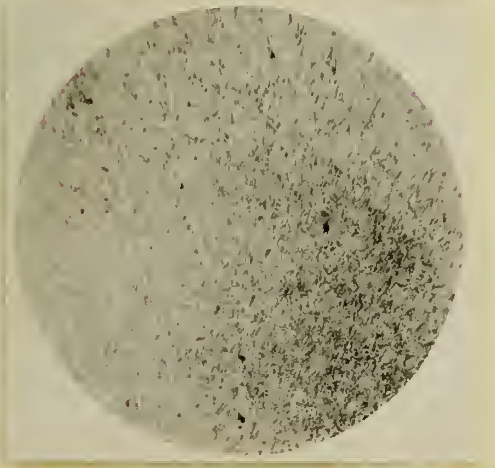


Fig.3b.
Steel B annealed.
Etched 50 Seconds.
Exposed 90 Seconds.
X 300.

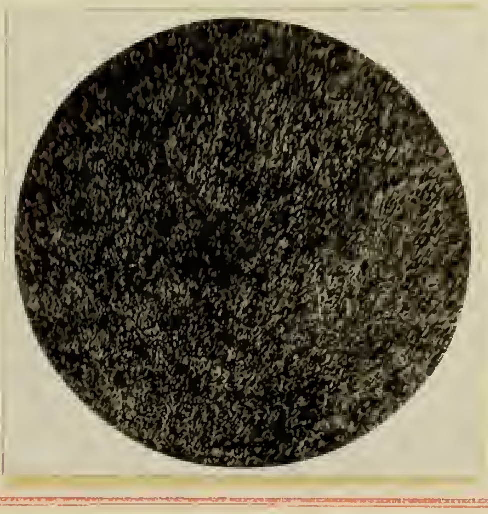


PLATE IX.

Steel A--Sample from Eng.Soc.. Steel B--From T.& A.M.Lab.

Fig. 6a.
Steel B quenched 800°..
Etched 100 Seconds.
Exposed 120 SEconds.
X 250.

Fig. 6b.
Steel B quenched 800°.
Etched 100 Seconds.
Exposed 120 Seconds.
X 300.

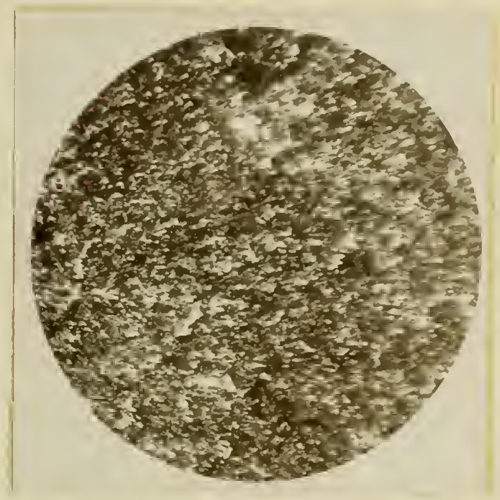


Fig. 5.
Steel B quenched 750°.
Etched 80 Seconds.
Exposed 105 Seconds.
X 250.

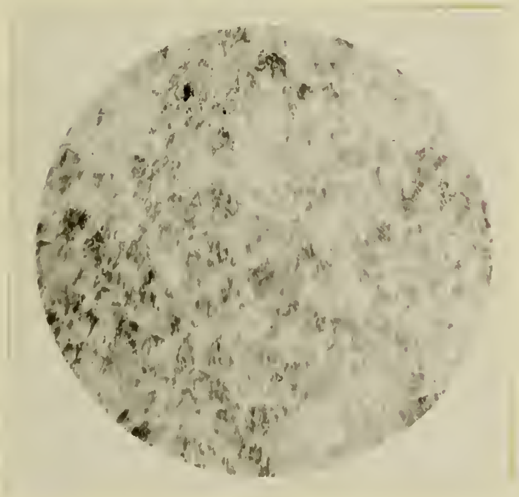


Fig. 7.
Steel B quenched 900°..
Etched 200 Seconds.
Exposed 120 Seconds.
X 250.

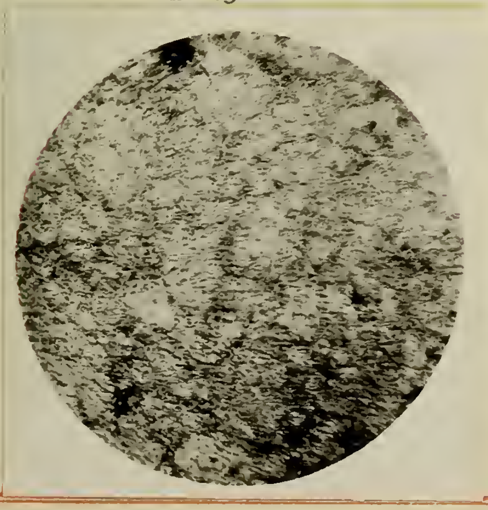
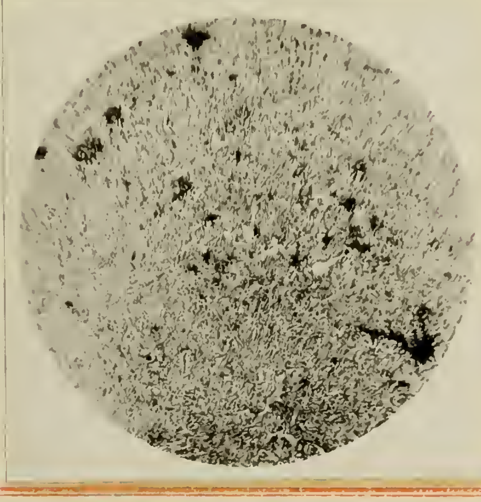


Fig. 8.
Steel B quenched 1000°.
Etched 200 Seconds.
Exposed 105 Seconds.
X 250.



darker pearlite.

Figs. 6A and 6B, Steel B. Quenched at 800° C. in ice water. A mixture of ferrite with unsegregated pearlite.

Fig. 7, Steel B. Quenched at 900° C. in ice water. Lighter areas a mixture of ferrite and pearlite, with darker areas of pearlite.

Fig. 8. Quenched at 1,000° C, in ice water. Martensitic structure with light areas of austenite, and darker patches showing formation of troostite.

Hardness Tests. Few properties of iron and steel are more important than that of hardness. Hardness is defined as that property of a body which resists penetration. In general a high hardness number indicates a metal of good wearing qualities. In the case of shafting a good wearing surface is of prime importance. The hardness test may also serve as an indication of brittleness and untrustworthiness which otherwise might be unsuspected. It is even possible to determine the carbon content, and the tensile strength of steel solely from the hardness number of the annealed steel.

The method used to determine the hardness was Brinell's ball impression test, which consists in the pressing of a hardened steel ball into the surface of the object under test, by means of a fixed load of 3,000 Kgr. for a fixed length of time (30 seconds). The diameter of the steel ball was 10mm. The dimensions of the impression thus obtained, form the basis for calculating the hardness number, which varies in direct

ratio with the hardness itself.

Table VI

Specimen	Dia. of Impression mm.	Treatment ° C	Hardness Numeral
1	5.3	Received	125
2	4.6	Annealed	169
3	4.3	Annealed	195
4	5.2	Compound heat treatment	128
5	3.6	Quenched 750°	285
6	3.4	Quenched 800°	321
7	3.1	Quenched 900°	387
8	2.8	Quenched 1000°	476

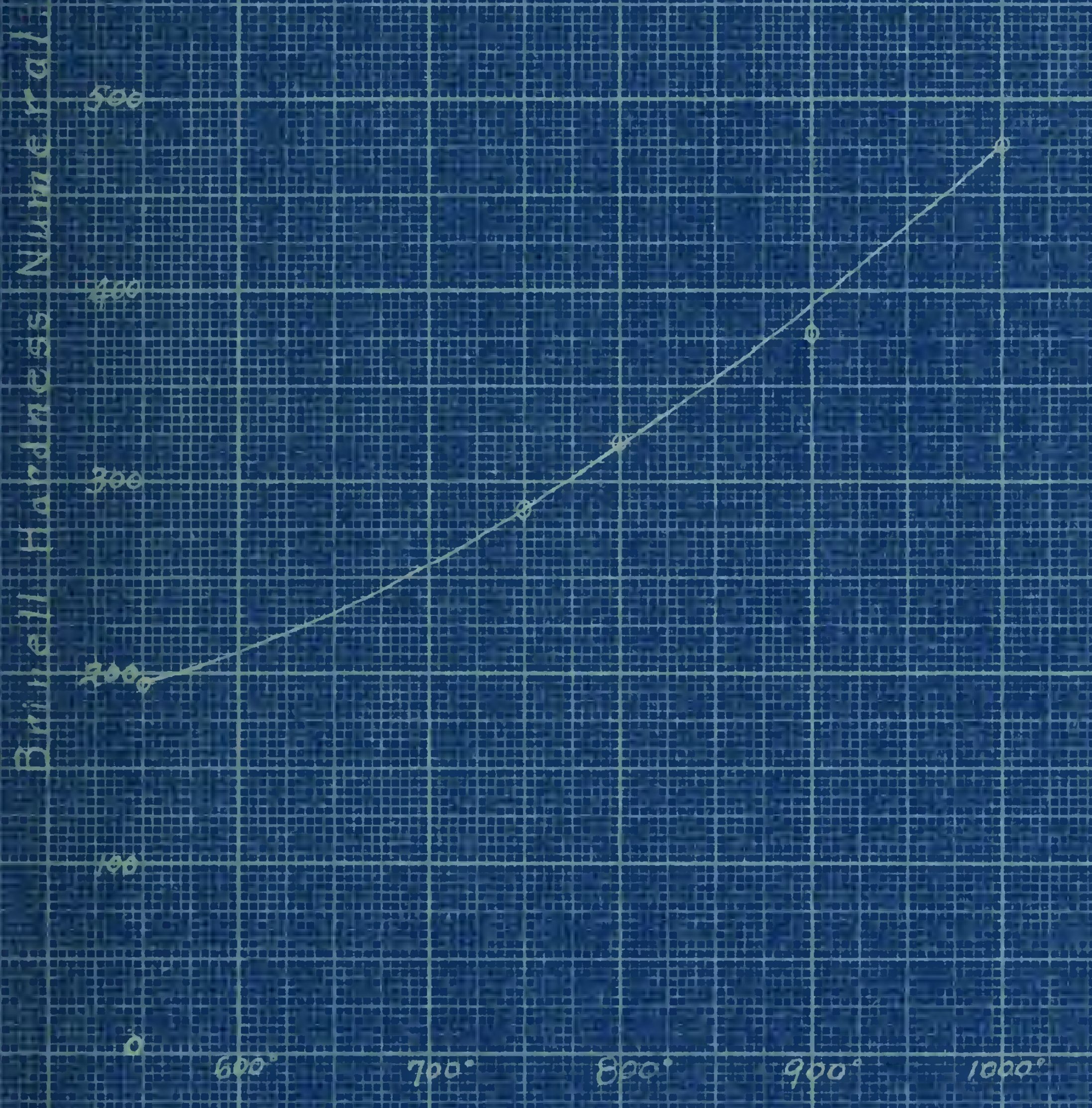
From the curve (Plate 10) we find that there must be a mathematical expression, which will give the relation of hardness to temperature at quenching, since the points nearly all lie on a smooth curve. Table 6 shows, as might be expected, that those specimens quenched at higher temperature have the greater hardness.

Failure of Material from Fatigue

It has been usual to describe a piece of wrought iron or steel which breaks in service with a bright crystalline fracture as having "become crystallized through fatigue". As yet there has been comparatively little written on the causes which ultimately produce fracture under fatigue stresses. Altho it might be thought that with the advent of microscopi-

PLATE X.

HARDNESS CURVE.



cal examination it would be a very simple matter to ascertain their effect, yet, with steel, considerable difficulty is experienced owing to the great purity and ductility of the ferrite crystals which do not readily show the effect of small strains.

As early as 1897 - 98 Mr. Thomas Andrews# in a series of articles published in Engineering writes that failures in steel appear to be produced by weakening the joints of the crystals, accelerated by cracks arising from minute flaws which exist in all commercial steels. This theory however did not meet with general acceptance.

Some time later Messrs. J. A. Ewing and J. C. W. Humfrey##, express their opinion that fracture is due to constant slipping in the crystals, which ultimately produces cracks in their cleavage planes.

In 1905 Mr. S. A. Houghton### writes that his observations show the action of stresses on the structure of the metal to be:

(a) Formation of slip bands, indicating slipping of the crystals

(b) Loosening of the joints between the crystals.

(c) Loosening of the particles of slag.

These changes result in the formation of cracks commencing as a rule from irregularities on the inner surface. These cracks are due to weakness in the cleavage planes of the

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The journal of the Iron and Steel Institute, No.1,1905 p. 383.

Philosophical Transactions, Vol. cc.

The Journal of the Iron and Steel Institute, No.1,1905.

crystals from continual slipping, and to a less degree to some loss of adhesion between the crystals. Some of the crystals appeared to have been broken up. The slag flaws seemed to have a restraining influence in the progress of the cracks.

One of the chief questions today which must be considered in the specifications of steel for shafting is that of the possibility of cold crystallization of iron and steel through fatigue. The manufacturer of steel often gives this as the reason for failure under stress, asserting that the material which has failed in service, was properly made and in perfect condition when it left his plant, and assuming that the failure must have been due to a subsequent deterioration caused by cold crystallization. This is a convenient excuse, and enables him to waive all responsibility, even when in most cases he knows the explanation is incorrect.

Mr. C. H. Ridsdale# says that while a coarse grain is more liable to yield to continual vibration, shock, or jar, yet there is no indication that such repeated stresses cause crystalline growth. On the other hand instances have been actually found where steel which had fractured after long vibration still had the same fine grain unchanged, as it had before being subjected to vibration.

Dr. J. E. Stead## writes that crystallization does not develop in cold iron under continued vibratory stresses.

\# The Journal of the Iron and Steel Institute, 1903, No.2, pp. 271.

\## The Journal of the Iron and Steel Institute, 1913, No.2.

Such stresses do however cause a weakening of the cohesion between the cleavage and joints of the crystals, so that after fatigue, a coarsely crystalline iron is much more easily fractured than before. Such material before fatigue will on breaking have a fibrous structure due to the crystals being drawn out. After fatigue however, it will give a crystalline fracture due to splitting along the cleavages of the iron.

Mr. F. Rogers# found that alternating stresses in iron and steel may or may not produce a crystalline fracture. At the same time the original unstressed specimen when broken by nicking and a blow had exactly the same crystalline surface, showing that the alternating stresses had not produced the crystalline structure in the final fracture.

It would seem therefore that if a piece of steel breaks in service with a crystalline fracture, it would have done likewise when new giving exactly the same crystalline structure. It also appears that repeated stresses will not give rise to crystalline structure in a material originally having a fibrous or silky fracture.

In fact there is no evidence that fatigue causes metals to become more coarsely or perfectly crystalline. Therefore there is no need when making specifications for shafting of considering its "becoming crystallized through fatigue", since repeated stresses cause no alternation in its structure

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other than the loosening of the crystal units which ultimately leads to direct destructive results.

Conclusion. The ultimate requirements of steel are that it shall satisfy the purpose for which it is intended. If it does this thoroughly it matters not what its composition is or how it has been treated provided it can be manufactured on an economic basis. The microphotographs prove that heat treatment alters the intermolecular structure, and even though specifications do determine the chemical composition, yet the physical tests or properties vary with different heat treatments. The heat treatment which was found to place steel for shafting in the best physical condition was to first anneal from above Ar_3, quench from above Ar_1, and finally temper from just below Ar_1. This treatment, while it does not produce the best wearing surface, was the only one studied which gave results satisfactorily fulfilling the requirements of the American Society for Testing Materials.